

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of
Ryuji Shiozaki et al.

Serial No.: 10/500,819

Group Art Unit: 1745

Filed: July 7, 2004

Examiner: Chuo, Tony Sheng Hsiang

For: POSITIVE ACTIVE MATERIALS AND NON-AQUEOUS ELECTROLYTE
BATTERIES EMPLOYING THE SAME

Honorable Commissioner of Patents
Alexandria, VA 22313-1450

DECLARATION UNDER 37 CFR 1.132

Sir:

Comes now the inventor Daisuke ENDO (hereinafter referred to as the "Declarant"),
and for his Declaration under 37 CFR 1.132, states and avers as follows:

1. That I graduated from Osaka City University, Graduate School of Engineering, Faculty of Engineering, Course of Applied Chemistry in March of 2002. I have been employed by YUASA CORPORATION since April of 2002. From 2002 to present, I have been engaged in research and development regarding active materials for lithium-ion batteries.
2. That I am the same Mr. Daisuke ENDO who performed the experimentation reflected in the declaration under 37 C.F.R. §1.132 filed September 25, 2008 in the present application, the same being signed by him on September 16, 2008.
3. That I have read and am familiar with the Sunagawa (U. S. Patent No. 6,040,090) which discloses a positive electrode material having the composition deemed as $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ (Table 5, Example 39), which the Examiner relies on to support his position.
4. That I have conducted experiments to investigate whether $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ disclosed by the Sunagawa reference as Example 39 in Table 5 shows a single phase

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structure belonging to space group R3-m as a result of examination by x-ray diffractometry. These experiments and the results of these experiments are described in Declarant's Exhibit 1 attached hereto and incorporated by reference herein.

5. That based on the results of my experimentation as illustrated in Declarant's Exhibit 1 attached hereto, I have concluded that the composition $\text{LiMn}_{0.03}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ disclosed by the Sunagawa reference as Example 39 in Table 5 clearly does not show a single phase structure belonging to space group R3-m as a result of examination by x-ray diffractometry.


I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application for any patent issued thereon.

Further Declarant sayeth naught.

July 29, 2009
Date

Daisuke Endo
Daisuke ENDO, Declarant

Prepared by:



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DECLARANT'S EXHIBIT 1

Experimentation

[Object]

It was an object of the experiment to prepare $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ disclosed by the Sunagawa reference as Example 39 in Table 5, in accordance with the methods disclosed therein and examine if the $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ "shows a single phase structure belonging to space group R3-m as a result of examination by x-ray diffractometry".

[Methods Employed by the Experiment]

While the Sunagawa reference discloses the method employed in Example 1 (column 6, lines 53-65) (hereinafter referred to as "Preparation Method A") and the method employed in Example 20 (column 7, line 66 to column 8, line 16) (hereinafter "Preparation Method B"), it is not clear which of the two methods was employed to prepare the $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ disclosed as Example 39 in Table 5. Both of the methods were, therefore, employed.

(Preparation according to Preparation Method A)

In the preparation of the positive electrode, particles of $\text{Ni}(\text{OH})_2$, $\text{Co}(\text{OH})_2$ and Mn_2O_3 having a mean particle size of $0.01\text{ }\mu\text{m}$ were added to $\text{LiOH}\cdot\text{H}_2\text{O}$ in a predetermined molar ratio and mixed together. The resultant mixture was heat treated in an atmosphere of dry air at 800°C . for 8 hours thereby to obtain a compound oxide of lithium and transition metals containing Li, Ni, Co and Mn in a molar ratio of $\text{Li}:\text{Ni}:\text{Co}:\text{Mn} = 1:0.05:0.9:0.05$. The resultant compound oxide was crushed to obtain a positive electrode material for each example and comparative example, which had a mean particle size of about $5\text{ }\mu\text{m}$.

(Preparation according to Preparation Method B)

In the preparation of a positive electrode, a mixture solution containing Ni, Co and Mn in a molar ratio of $\text{Ni}:\text{Co}:\text{Mn}=0.05:0.9:0.05$ was prepared by agitating nickel sulfate, cobalt sulfate and manganese sulfate in an agitating vessel. The resultant mixture solution was further agitated with an aqueous solution of sodium hydroxide gradually added thereto whereby a hydroxide of Ni, Co and Mn was co-precipitated. Then, the compound hydroxide of these metals was mixed with $\text{LiOH}\cdot\text{H}_2\text{O}$. At this time, a molar ratio of Li to the sum of the metal elements contained in the metal compound hydroxide was 1:1. Subsequently, the resultant mixture product was heat treated in the atmosphere of air at 800°C . for 8 hours to obtain a lithium-metal compound oxide.

The X-ray diffraction pattern of the sample prepared by each method was obtained by the powder X-ray diffraction method. The results are shown below in Figs. 1 and 2. Fig. 1 shows the X-ray diffraction pattern of the sample prepared by Preparation Method A, while Fig. 2 shows the X-ray diffraction pattern of the sample prepared by Preparation Method B.

Both of Figs. 1 and 2 show not only the peaks belonging to R3-m, but also the peaks not belonging to R3-m as shown by arrows. This confirms that $\text{LiMn}_{0.05}\text{Ni}_{0.05}\text{Co}_{0.9}\text{O}_2$ disclosed by the Sunagawa reference as Example 39 in Table 5 does not "show a single phase structure belonging to space group R3-m as a result of examination by x-ray diffractometry", and certainly does not "consist essentially of a single phase structure belonging to space group R3-m". Fig. 3 below is an enlargement of a part of Fig. 2.

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Fig. 1

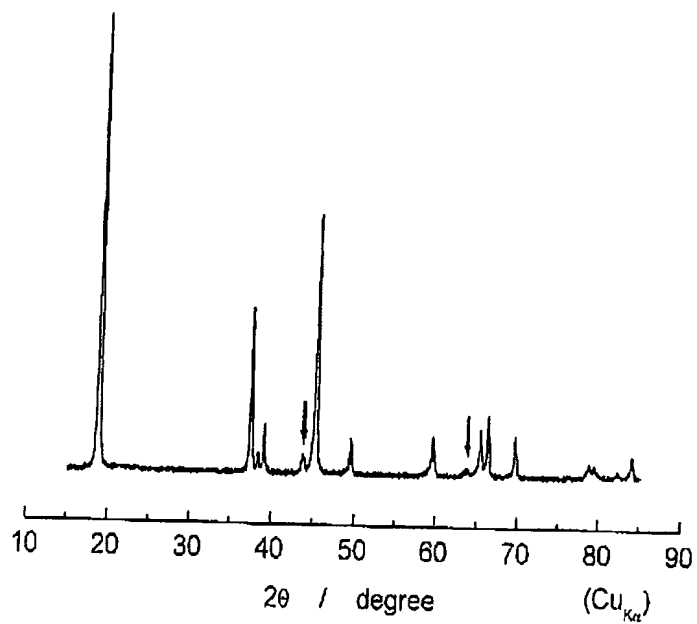
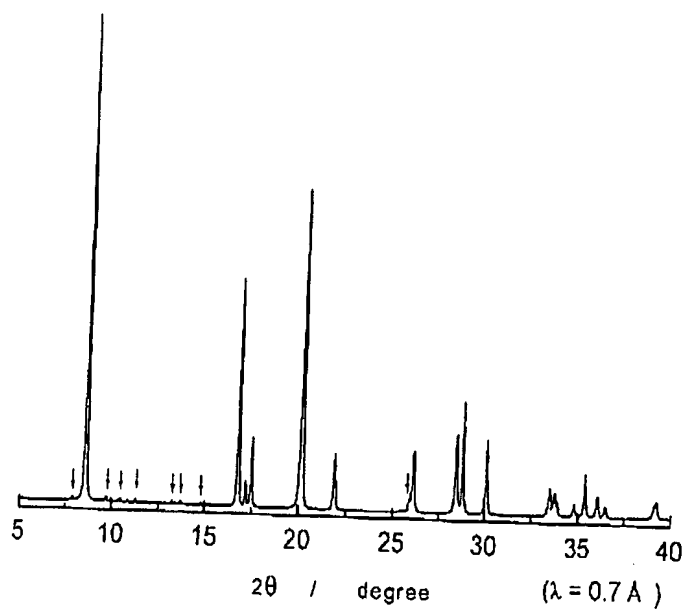


Fig. 2



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Fig. 3

